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Research on the grain growth and the mechanism of (U,Ti)O_2 dispersion fuel microspheres

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Abstract

This paper studies the preparation of UO_2 dispersion fuel microspheres with 0.3 wt% of doped titanium by the sel–gol method. The experimental results show that between 1250 °C and 1450 °C, grain growth speeds are different with a rapid growth of some grains and a "double peaks" feature of grain sizes distribution, while at 1550 °C, the grain sizes have normal distribution with the average grain size of about 42 μm. Based on the calculation, between 1250 and 1550 °C, the average activation energy of the grain growth of (U,Ti)O_2 microspheres, Ea, was 232.79 kJ/mol, which is significantly lower than that of the UO_2 microspheres. In the later sintering period of (U,Ti)O_2 microspheres, U_4O_9 is formed in some parts of the microspheres, accelerating the U-atom diffusion rate and the grain growth.

Keywords: B. Grain size; UO_2 microspheres; Activation energy; Dispersion fuel

1. Introduction

In order to improve the efficiency of nuclear power, alleviate the handling and disposal pressure of spent fuel, developing fuel elements with high burnup and a long life is a long-term goal for nuclear power plants. Compared with the UO_2 ceramic pellets rod fuel elements used in the current water reactor power plants, the fuel particles dispersing in metal matrix form metal–ceramic dispersion fuel (Fig.1), a characteristic of low core temperature, inherent high safety, high radiation resistance, deep burn-up, long service life etc. [1–6], thus making it have broad application prospects in water power reactors also [7–9]. Metal–ceramic dispersion fuel is made up of the fuel phase and matrix phase. Nuclear fuel particles are evenly distributed in the base material. As far as fuel phase particles are concerned, the ideal dispersion fuel phase should have the following advantages: (1) the spherical particle; (2) higher relative theoretical density and mechanical strength; and (3) good microstructures. Therefore, it is more complex and difficult to prepare than the UO_2 pellets in nuclear power rod typed fuel element.

Although metal–ceramic dispersion nuclear fuel combines the advantages of ceramic fuel and metal fuel [10], overcomes the shortcomings of each fuel, thus improving the irradiation stability and increasing burnup depth, the pure UO_2 fuel dispersion phase has been difficult to meet higher requirements. Because the further deepening of burnup can lead to sharp increase in release rate of fuel fission products, UO_2 fuel irradiation swelling will be worse [11–15]. So it has become an important research direction to add other metal elements to UO_2 to inhibit fission gas release and improve the irradiation resistance and corrosion resistance of fuel phase so as to improve the fuel burnup [16,17].

At present, (U,Ti)O_2 fuel spheres prepared by the sol–gel process are considered good ceramic fuel, which can improve fuel burnup and irradiation stability under high burnup. The microstructure of UO_2 has a great influence on the irradiation behavior inside the reactor. Therefore, in order to guarantee the stable safe running of fuel elements in the reactors, there are certain requirements for the microstructure of UO_2, especially the fuel grain size.

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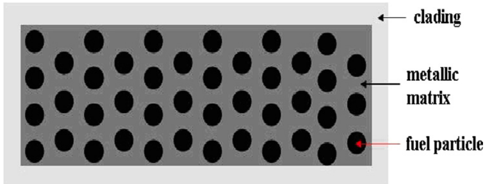

Fig. 1. Metal–ceramic composite dispersion fuel.

The larger the grain size of UO_2 pellets, longer the fission gas migration distance and lower the fission gas release rate. Lowering the pellets-cladding interaction (PCI) can significantly improve the safety margin of the fuel in the reactor in the process of running. In early experimental research work, we found that $(U,Ti)O_2$ fuel microspheres can be prepared with uniform distribution of titanium by using the sol–gel method [18]. A small amount of doped titanium can effectively promote the grain growth of UO_2 microspheres. In the same sintering conditions, the grain size of $(U,Ti)O_2$ fuel microspheres is much bigger than that of the UO_2 microspheres without doped titanium. The grain growth behavior between $(U,Ti)O_2$ fuel microspheres and UO_2 microspheres is definitely different; so the study on the grain growth mechanism of $(U,Ti)O_2$ fuel microspheres has certain theoretical guiding significance and academic value for improving and controlling the quality of $(U,Ti)O_2$ fuel microspheres, optimizing the process parameters as well as doing research on the dispersion nuclear fuel with high intrinsic safety in a water power reactor. Based on in-depth research and analysis about the grain growth of $(U,Ti)O_2$ fuel microspheres, the distribution of different grain sizes, microstructure morphology, titanium distribution inside the microspheres and grain growth kinetics, etc the grain growth mechanism of $(U,Ti)O_2$ fuel microspheres is discussed.

2. Experimental method

2.1. Preparation of $(U,Ti)O_2$ microspheres

The doping content of titanium is fixed as 0.3 wt%. The sol–gel method is used to prepare ADUN sol containing titanium. Take a certain amount of acid, deionized water titanium compound ADUN and mix them in the beaker and prepare transparent titanium-doped ADUN sol after uniform stirring for a period of time. Through sol dispersion, gelling, washing, calcination, reduction sintering and other processes titanium-doped ADUN sol will change to $(U,Ti)O_2$ fuel microspheres. The sintering atmosphere is H_2 and the sintering temperatures are 1250 °C, 1350 °C, 1450 °C and 1550 °C. The sintering time is 4 h.

2.2. Analysis and measurement

After the sintering of $(U,Ti)O_2$ fuel microspheres, sand paper is used to gradually grind (until 800 mesh) the samples of microspheres. Cr_2O_3 turbid liquid is used for mechanical polishing. The volume fraction of 50% concentrated $HNO_3+50\%$ H_2O with high purity is used for chemical etching for 1～3 min to reveal all the grain boundaries for metallographic observation and image acquisition. Image instrument is used for statistical measurement of the mean grain size. Scanning electron microscopy (SEM) and energy dispersive spectrometer (EDS) are used to observe the microstructures of $(U,Ti)O_2$ fuel microspheres, titanium element distributions as well as the titanium content on the grain boundary at different sintering temperatures.

3. Discussion of grain growth mechanism of $(U,Ti)O_2$ fuel microspheres

3.1. Calculation of grain growth activation energy of $(U,Ti)O_2$ fuel microspheres

For general material, the grain growth conforms to the classic Arrhenius formula:

$$k = A\exp\left(-\frac{E_a}{RT}\right) \tag{1}$$

In the formula, k is grain growth speed, A refers to the pre-exponential factor, E_a is grain growth activation energy, R refers to the gas constant and T is the heat treatment temperature.

If $k=dG/dt$, and k is a constant within a certain temperature range, integral calculation on both sides goes like

$$\int_{t_1}^{t_2} k\,dt = \int_{G_1}^{G_2} dG \Rightarrow k = \frac{G_2-G_1}{t_2-t_1} \tag{2}$$

That is $\left((G_2-G_1)/(t_2-t_1)\right) = A\exp\left(-\left(E_a/RT\right)\right)$, take logarithm on both sides to get:

$$\ln\left(\frac{G_2-G_1}{t_2-t_1}\right) = \ln A - \frac{E_a}{RT} \tag{3}$$

Between the 2 temperatures T_1 and T_2, the average activation energy is

$$E_a = \frac{RT_1T_2}{T_1-T_2}\ln\left(\frac{G_2-G_0}{G_1-G_0}\right) \tag{4}$$

In the formula, G_1 and G_2 are the average grain sizes at the temperatures of T_1 and T_2.and G_0 is the average grain size of samples before annealing. $(U,Ti)O_2$ fuel microspheres in the present study keep thermal preservation for 4 h at 1250 °C, 1350 °C, 1450 °C and 1550 °C respectively with the average grain sizes of 3.3 μm, 6.8 μm, 25.4 μm and 41.9 μm (Fig. 2). According to the formula (4), the average grain growth activation energy of $(U,Ti)O_2$ fuel microspheres, E_a, can be calculated between 1250 °C and 1550 °C. Calculation results are listed in Table 1.

According to Table 1, between 1250 °C and 1550 °C, the average grain growth activation energy of $(U,Ti)O_2$ fuel microspheres, $E_a=(181.72+361.7+154.95)/3=232.79$ kJ/mol.

The average grain growth activation energy of UO_2 microspheres $E_{a0}=518.32$ kJ/mol. Nichols [19] proposed a phenomenological dynamic equation for the normal grain growth (5), which can also be applied to the grain growth kinetics when the second phase impurities are present at the same time.

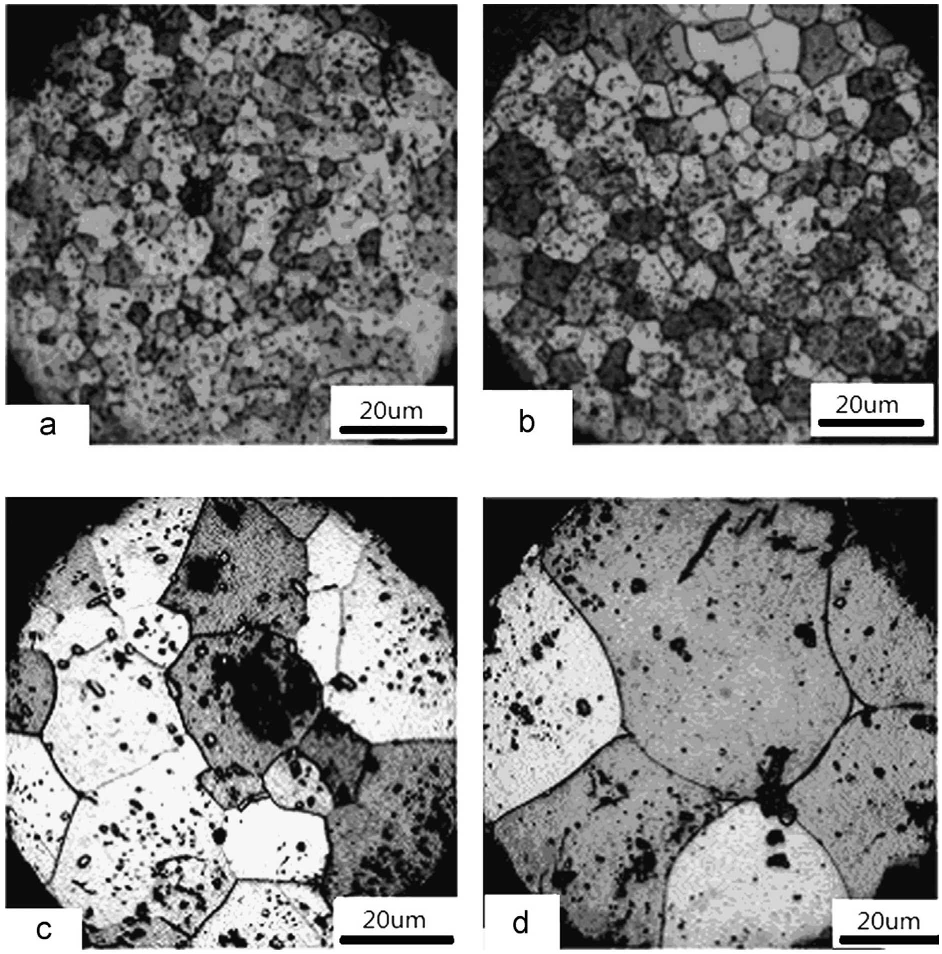

Fig. 2. Metallographic photos of 0.3 (wt%) titanium-doped UO_2 microspheres at different sintering temperatures.

Table 1
The grain growth activation energy of titanium-doped UO_2 microsphere.

Temperature range (°C)	G_1 (μm)	G_2 (μm)	G_0 (μm)	E_a (kJ/mol)
1250–1350	3.3	6.8	0.56	181.72
1350–1450	6.8	25.4	1.22	361.70
1450–1550	25.4	41.9	3.16	154.95

According to the formula (5) the cube of the grain size after sintering increases with the decrease of the grain growth activation energy Q. Because the grain growth activation energy of UO_2 microspheres $E_a = 232.79$ kJ/mol $< E_{a0} =$ 518.32 kJ/mol, and there is a dramatic fall compared to the UO_2 microspheres without doped titanium, the grains of titanium-doped UO_2 microspheres grow more easily.

$$G^3 - G_0{}^3 = k\exp\left(-\frac{Q}{RT}\right)t \quad (5)$$

3.2. The effect of sintering temperatures on the average grain size and distribution of $(U,Ti)O_2$ fuel microspheres

Fig.3 shows that a small amount of titanium can effectively promote the grain growth of UO_2 microspheres. When the sintering temperature is above 1350 °C, the grain sizes of

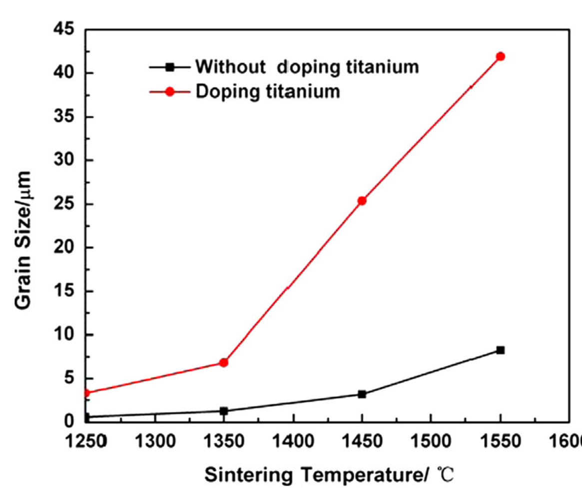

Fig. 3. The influence of sintering temperatures on the grain sizes of $(U,Ti)O_2$ fuel microspheres.

(U,Ti)O_2 fuel microspheres go up to a parabolic rise with the increase of temperatures. Although the grain sizes of UO_2 microspheres also increase with increasing temperature; the growth rate is far lower than that of the (U,Ti)O_2 fuel microspheres. When the sintering temperature is 1250 °C, (U,Ti)O_2 fuel microspheres grain size is very small but uniform with the average size of 3.3 μm(Fig.4). At the sintering temperature of 1350 °C, the average grain size is 6.8 μm. But part of the grains in the (U,Ti)O_2 fuel microspheres begin to grow rapidly with the maximum of 20.0 μm, which is about 7 times the size of the average grain size of (U,Ti)O_2 fuel microspheres at the temperature of 1250 °C, and the minimum grain size is only 2.5 μm, The grain size distribution in Fig.5 shows a "double peaks" phenomenon with the sintering density of microspheres being about 96.5% TD. Therefore, it can be considered that grain growth occurs mainly after the microspheres densification. At the sintering temperatures of 1450 °C and 1350 °C, the phenomenon is almost similar. Part of the grains of the (U,Ti)O_2 fuel microspheres grow rapidly, and grain sizes differ more dramatically with the largest grain size of about 50.5 μm, 8 times as large as the average grain size at the temperature of 1350 °C and the smallest grain size of 8.0 μm. The grain size distribution still presents a "double peaks" phenomenon (Fig.6). Through the above-mentioned analysis, it shows that when the sintering temperature is between 1250 °C and 1250 °C, part of the grains remain in rapid growth and eventually become big in size, but the small grains grow slowly within this temperature range. When the sintering temperature is 1550 °C, the grain sizes are uniform, with the maximum of about 65.0 μm and a minimum of about 11.0 μm. According to the grain size distribution in Fig.7, the "double peaks" phenomenon has disappeared with the average grain size of about 41.9 μm and the average grain size of pure UO_2 microspheres after being sintered at 1550 °C is about 8.3 μm. Therefore, doping titanium can significantly promote the grain growth of UO_2 microspheres. Figs. 8 and 9 show SEM images of (U,Ti)O_2 fuel microspheres at different sintering temperatures. The grain growth and evolution of(U,Ti)O_2 fuel microspheres can be observed between 1250 °C and 1550 °C.

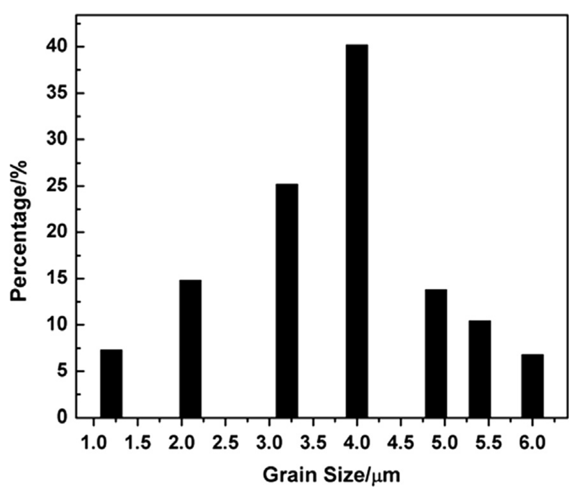

Fig. 4. The grain sizes distribution of (U,Ti)O_2 fuel microspheres at 1250 °C.

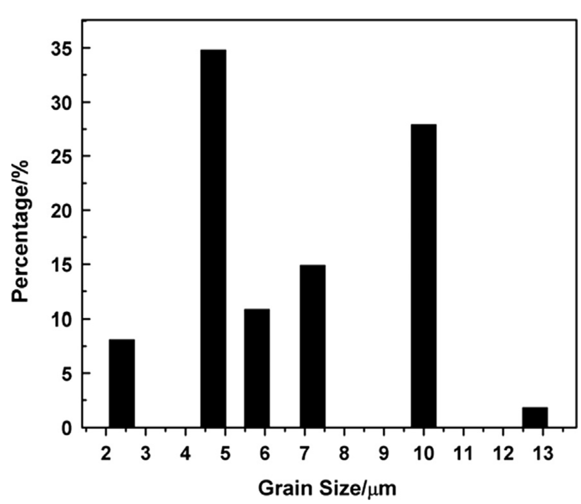

Fig. 5. The grain sizes distribution of (U,Ti)O_2 fuel microspheres at 1350 °C.

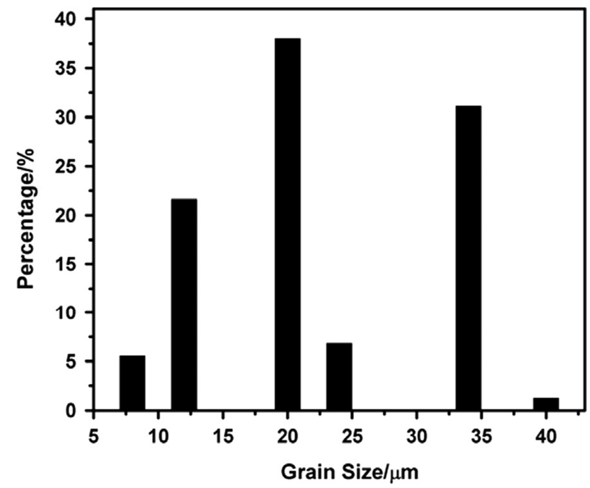

Fig. 6. The grain sizes distribution of (U,Ti)O_2 fuel microspheres at 1450 °C.

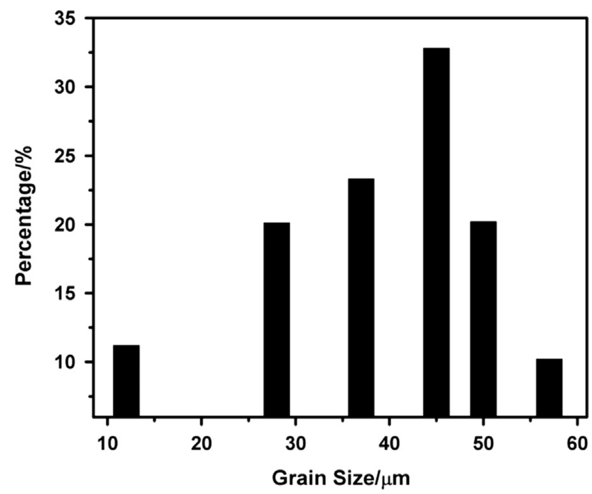

Fig. 7. The grain sizes distribution of (U,Ti)O_2 fuel microspheres at 1550 °C.

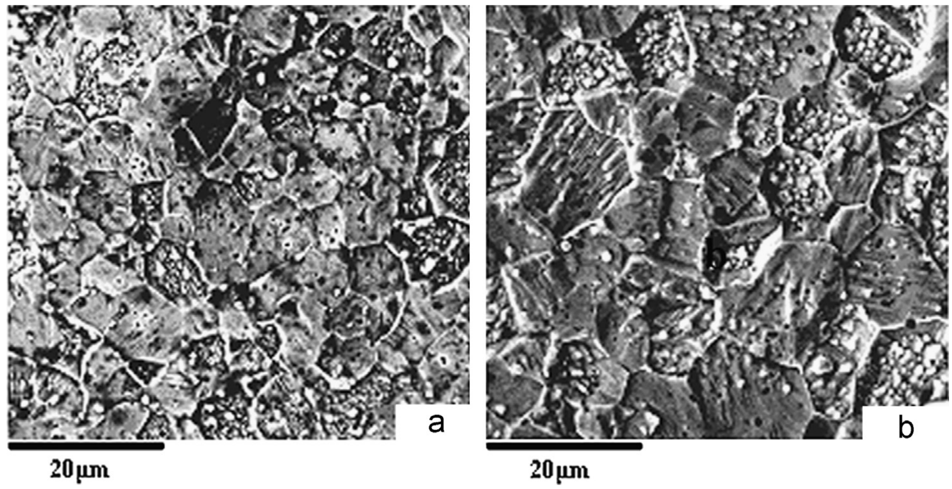

Fig. 8. SEM photos of $(U,Ti)O_2$ fuel microspheres ((a) 1250 °C; (b) 1350 °C).

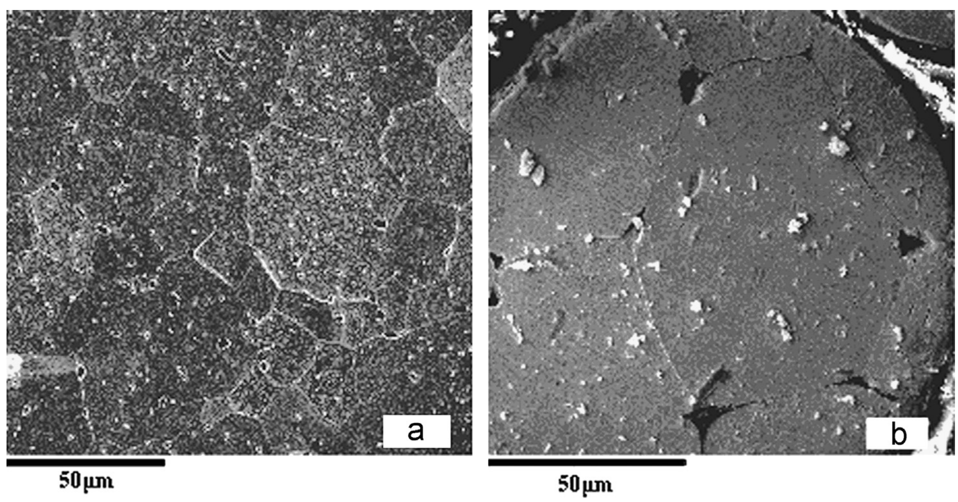

Fig. 9. SEM photos of $(U,Ti)O_2$ fuel microspheres ((a) 1450 °C; (b) 1550 °C).

3.3. The effect of doped titanium on oxygen potential in the sintering process of UO_2 microspheres

The addition of TiO_2 has a great effect on the oxygen potential in microspheres in the process of sintering. Titanium oxide comes in a wide variety, including Ti_2O, TiO, Ti_2O_3, TiO_2, etc. At high temperature in the H_2 atmosphere, TiO_2 might release oxygen by changing its valence state and. TiO_2 exists in the form of TiO_{2-x}.

$$TiO_2 = TiO_{2-x} + xO \quad (6)$$

In TiO_{2-x}, x is mainly related to the sintering temperature. According to the literature [20] which describes the relationship between the temperature and oxygen potential of TiO_2, when the sintering temperature is 1250 °C, the oxygen potential of TiO_2 is very low in microspheres, which leads to the presumption that TiO_2 emits very little oxygen that is not enough to make UO_2 produce new phases. As the sintering temperature continues to increase, the oxygen potential of TiO_2 in microspheres rises rapidly. When the temperature reaches 1500 °C, the oxygen potential of TiO_2 is more than that of H_2+5 vol%H_2O. TiO_2 has a high oxygen potential and the deviation of TiO_{2-x} from the stoichiometric result is larger with more oxygen release, which may cause the generation of high U_4O_9 phase in titanium-doped UO_2 microspheres.

From what has been discussed above, with the rise of sintering temperatures, after the addition of a small amount of TiO_2 in UO_2 microspheres, titanium oxide constantly releases oxygen. Through the joint action of TiO_2, some phase composition changes take place in UO_2 titanium-doped microspheres between 1250 °C and 1550 °C. Only a small amount of UO_2 phase could change to the U_4O_9 phase, but because of the small amount of doped titanium, the proportion of U_4O_9 was very little as well.

3.4. The effect of doped titanium on grain growth of UO_2 and U_4O_9 phase in UO_2 microspheres

Because of the small amount of doped titanium in titanium-doped UO_2 microspheres, and smaller atomic radius of Ti than atomic radius of U, the grain growth velocity in titanium-doped UO_2 microspheres mainly depends on the diffusion velocity of U, D_U. If the grain diameter is G and the grains grow at a certain temperature as equiaxial grains, from t_0 to t the growth volume of grains ΔV is as follows:

$$\Delta V = \frac{\pi}{6}(G_t{}^3 - G_{t_0}^3) = D_U(t - t_0) \quad (7)$$

According to the research system for titanium-doped UO_2 microspheres and Formula (7), the ratio of the growth volume of grains between U_4O_9 phase and UO_2 phase in a certain period of time of$(t_0 - t)$ is

$$\frac{G^3{}_{(U_4O_9)t} - G^3{}_{(U_4O_9)t_0}}{G^3{}_{(UO_2)t} - G^3{}_{(UO_2)t_0}} = \frac{D_{U_4O_9}}{D_{UO_2}} \quad (8)$$

Since the initial grain size is very small, $G^3{}_{(UO_2)t} \geq G^3{}_{(UO_2)t_0}$ and $G^3{}_{(U_4O_9)t} \geq G^3{}_{(U_4O_9)t_0}$, $G^3{}_{(U_4O_9)t_0}$ and $G^3{}_{(UO_2)t_0}$ can be ignored. Formula (8) goes as follows

$$\frac{G^3{}_{(U_4O_9)t}}{G^3{}_{(UO_2)t}} = \frac{D_{U_4O_9}}{D_{UO_2}} \quad (9)$$

Predecessors have done a lot of research on U atom diffusion coefficient; the research conclusions in the literature [20] are widely recognized, its content is as follows:

$$D_{UO_2} = 0.19\exp\left(-\frac{Q_1}{KT}\right) \quad (10)$$

$$D_{UO_{2+x}} = 1.33X^2 \exp\left(-\frac{Q_2}{KT}\right) \quad (11)$$

The average grain boundary activation energy of UO_2,, $Q_1 = 377.6$ kJ/mol. The average activation energy of UO_{2+X}, $Q_2 = 276.3$ KJ/mol. $X = 0.25$ in Formula (11).

At the sintering temperature of 1350 °C, based on Formulas (10), (11) and (9), the following calculation can be obtained:

$$\frac{G^3{}_{(U_4O_9)}}{G^3{}_{(UO_2)}} = 796.22, \quad \frac{G_{(U_4O_9)}}{G_{(UO_2)}} = 9.3$$

At the sintering temperature of 1450 °C, based on Formulas (10), (11) and (9), the following calculation can be obtained:

$$\frac{G^3{}_{(U_4O_9)}}{G^3{}_{(UO_2)}} = 515.0, \quad \frac{G_{(U_4O_9)}}{G_{(UO_2)}} = 8.0$$

At the sintering temperature of 1550 °C, based on Formulas (10), (11) and (9), the following calculation can be obtained:

$$\frac{G^3{}_{(U_4O_9)}}{G^3{}_{(UO_2)}} = 343.0, \quad \frac{G_{(U_4O_9)}}{G_{(UO_2)}} = 7.0$$

From Fig. 1, at 1250 °C the grain sizes in (U,Ti)O_2 fuel microspheres are uniform with the average grain size of ~3.3 μm. At 1350 °C the largest grain size in (U,Ti)O_2 fuel microspheres is 20.0 μm, which is about 7 times the size of the average grain size of (U,Ti)O_2 fuel microspheres at the temperature of 1250 °C. At 1450 °C the largest grain size in (U,Ti)O_2 fuel microspheres is ~50.5 μm, which is about 8 times the size of the average grain size of (U,Ti)O_2 fuel microspheres at the temperature of 1350 °C. At 1550 °C the maximum grain size in (U,Ti)O_2 fuel microspheres is 65 μm, which is about 2.3 times the size of the average grain size of (U,Ti)O_2 fuel microspheres at the temperature of 1450 °C, which is quite diverse from the above calculation of seven times. Therefore, it is basically reasonable to believe that between 1250 °C and 1450 °C excess oxygen provided by doped titanium generates U_4O_9 phase in the microspheres. Due to differences in diffusion velocity, U_4O_9 phase becomes big grain areas and the UO_2 phase becomes small grain areas. The growth speed of big grains is higher than that of the small grains and small grains around the big grains disappear continuously. Although as a whole they are continuously growing, the speed is slow. When the small grains are covered with big grains and the grain boundary changes from the curve into a straight line, the grain growth driving force disappears and the grain growth almost ends. At the same time, U_4O_9 is reduced to UO_2 if (U,Ti) O_2 fuel microspheres are sintered at rising sintering temperatures in reducing atmosphere H_2 after a long time.

Therefore, for (U,Ti)O_2 fuel microspheres in late sintering (above 1250 °C), the main cause of rapid grain growth possibly lies in the fact that titanium provides excess oxygen through valence changes, generating U_4O_9 with a high state which promotes diffusion velocity of U atom and leading to the larger grain sizes in (U,Ti)O_2 fuel microspheres than those in the UO_2 microspheres.

4. Conclusion

1. According to the calculation, between 1250 °C and 1550 °C the average grain growth activation energy of (U,Ti)O_2 fuel microspheres $E_a = 232.79$ kJ/mol, significantly lower than that of the non-doped titanium UO_2 microspheres ($E_{a0} = 518.32$ kJ/mol). Therefore, doping with titanium contributes to grain growth in the microspheres.
2. At sintering temperatures between 1250 °C and 1450 °C the grain growth speeds of (U,Ti)O_2 fuel microspheres are inconsistent. Part of the grains grow rapidly and eventually become big in size, while the small grains grow slowly within this temperature range. The grain size distribution shows "double peaks". When the sintering temperature is 1550 °C, the grain sizes are normally distributed.
3. When (U,Ti)O_2 fuel microspheres are sintered at 1250 °C, 1350 °C and 1450 °C, titanium is evenly distributed in the microspheres; titanium oxide is mostly distributed uniformly inside the microsphere grains and a small amount of titanium oxide precipitates and enriches on the grain boundary. But at the sintering temperature of 1550 °C, most of the titanium oxide enriches on the grain boundary.
4. (U,Ti)O_2 fuel microspheres in late sintering (above 1250 °C), the main cause of rapid grain growth possibly lies in the fact that titanium provides excess oxygen through valence changes, generating U_{4O9} with a high state in some parts of the microspheres which promotes diffusion velocity of U atom.

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